Fabrication of microstructures on photosensitive glass using a femtosecond laser process and chemical etching

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In this study a method for the fabrication of microstructures on the surface of and inside photosensitive glass by femtosecond laser-induced modification was developed. This technique was followed by heat treatment to crystallize the modified area, and the specimen was then placed in 8% HF acid solution for chemical etching. The results demonstrated that the etching time, scanning speed, and laser power are important parameters in the fabrication of microstructures and inner structures on photosensitive glass. In future, inner microstructures could be applied in the fields of biomedicine and cell detection.

Keywords: femtosecond laser, microstructures, photosensitive glass, etching time

1. Introduction

As photosensitive glass has unique optical transparency, hardness, chemical, and thermal resistance properties, it is a promising material for application in micro-fluidics and micro-optics. Microstructures have been fabricated on a photosensitive glass surface by femtosecond laser-induced modification [1–3]; this is followed by heat treatment to crystallize the modified area, and the microstructures are then placed in 8% HF acid solution for chemical etching. After the heat treatment process, the modification area crystallizes, and the etching time is shorter in the area of crystallization than in the amorphous area because the area is 20–40 times more soluble in HF acid solution [4–5]. The surface was selectively etched, resulting in the formation of fabricated microstructures. With a constant chemical etching time of 50 min, the diameters of the micro holes remained constant (at about 77 μm) with varying scanning speed [6]. The authors inferred that the irradiation area had received the exposure necessary for subsequent crystallization during heat treatment; however, the reason for the exposure area being much larger than the focus spot diameter (about 5 μm, as calculated using a numerical aperture 0.28 microscopic objective lens) was not further investigated.

The results of this study suggested that, given appropriate selection of irradiation parameters, microstructures can be fabricated on the surface of photosensitive glass in a short etching time and on the inside of photosensitive glass in a long etching time.

2. Experimental

The experiments were performed using a commercial photosensitive glass (Foturan, Schott Company) and a regenerative amplified mode-locked Ti:sapphire laser (SPIT FIRE, Spectra-Physics) with a low repetition rate of 1 kHz, a pulse duration of ~120 fs, a central wavelength of 800 nm, and a maximum pulse energy of ~3.5 mJ.

Figure 1 presents a schematic illustration of the experimental setup. In order to adjust the energy of the laser beam, the linear polarized Gaussian beam emitted from the laser was attenuated by a rotatable half-wave (λ/2) plate and a polarizing beam splitter (PBS). The transmitted component of the laser beam was incident upon a beam splitter (BS), the reflected beam was launched into a power detector, and the laser irradiation energy was measured. Meanwhile, the transmitted linearly-polarized laser beam was passed through a shutter and a series of reflective mirrors, subsequently entering a 10x objective lens (numerical aperture 0.26, M Plan Apo NIR, Mitutoyo) in the normal direction on the surface of the glass. The position of the objective lens was adjusted in the vertical (i.e., Z-axis) direction, and the femtosecond laser focused spot diameter was about 5 μm.

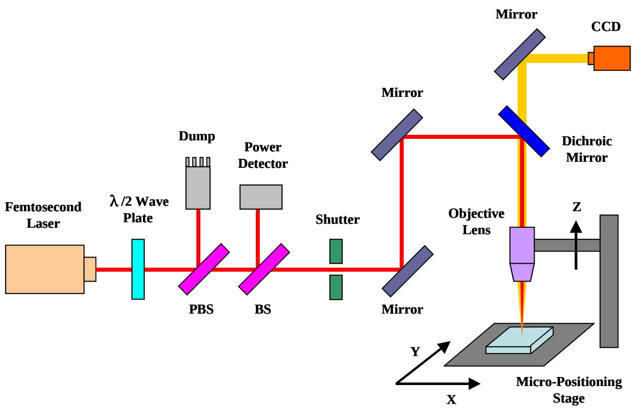

Fig. 1 Schematic illustration of the femtosecond laser system.

Microstructures were produced by translating the sample using an X–Y stage operating under the control of a PC-based micro-positioning system with an accuracy of less than 1 μm. The fabrication process was monitored continuously using a charge-coupled device (CCD) camera.

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After the laser-induced crystallization process, the Foturan glass was first heated up to 500°C at a rate of 5°C per minute, and after being maintained at this temperature for about one hour, the temperature was then ramped up to 600°C at a rate of 3°C per minute, and the Foturan glass held at this temperature for about one hour. Finally, the sample was cooled to room temperature under ambient conditions. After the heat treatment process, the samples were immersed in an 8% hydrofluoric acid (HF) etchant in an ultrasonic bath at room temperature for different etching durations.

3. Results and Discussion

Figure 2 presents microscope images showing the line patterns fabricated by setting the femtosecond laser system at a scanning speed of 0.05 mm/s and a laser power of 0.23–0.33 mW. Note that the specimen is in an unheated and unetched condition. It can be seen that the surface within the laser-irradiated area was slightly clearer than that within the unirradiated region. From Fig. 2(b)–(d), an obvious trench can be observed in the irradiated area, as the laser power is greater than the ablation threshold. No obvious surface modifications can be observed in Fig. 2(a).

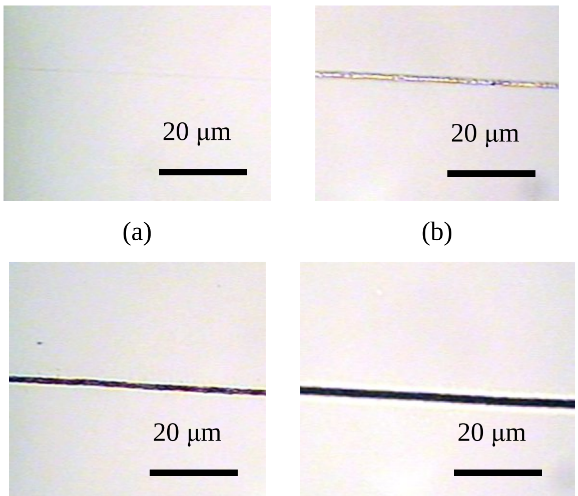


Fig. 2 Microscope images of irradiated Foturan glass under different laser powers before heat treatment and etching: (a) 0.23 mW, (b) 0.27 mW, (c) 0.30 mW, and (d) 0.33 mW.

The irradiated sample was then heated, leaving a clear line pattern on the glass substrate surface, as shown in Fig. 3(a)

The sample was then further etched for 50 min. Figure 4 shows SEM images of the line patterns, from which it can be seen that in every case the lines were of a width of around 45 μm.

Figure 5 is a plot of the variation of the measured line depth and width as a function of the scanning speed at a laser power of 0.122 mW. It was observed that the line width was constant at about 47 μm and the line depth decreased with increasing scanning speed.

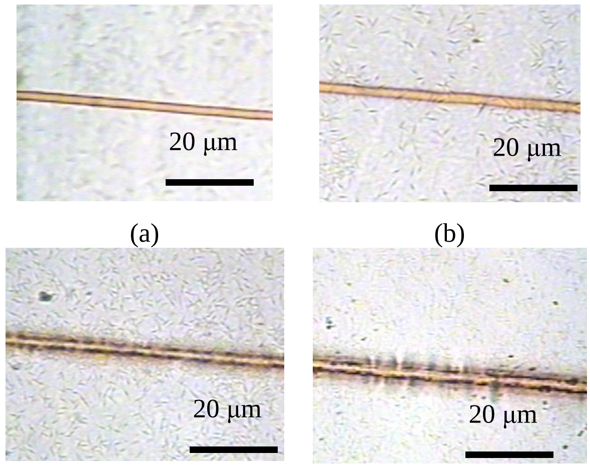


Fig. 3 Microscope images of irradiated Foturan glass after heat treatment and before etching: (a) 0.23 mW, (b) 0.27 mW, (c) 0.30 mW, and (d) 0.33 mW.

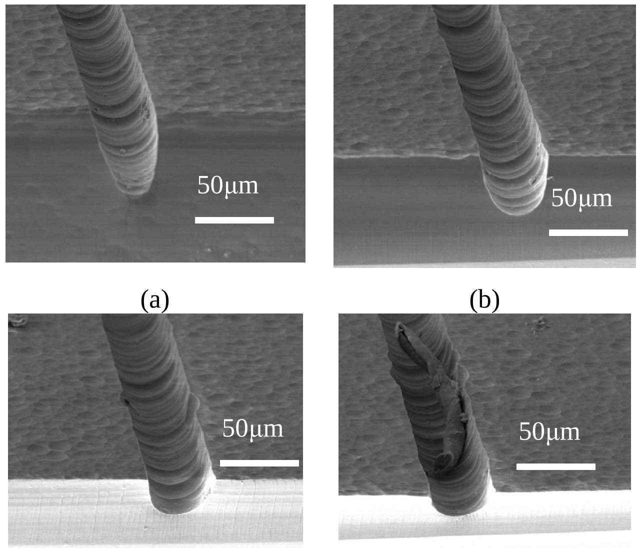


Fig. 4 SEM images of irradiated Foturan glass after heat treatment and etching: (a) 0.23 mW, (b) 0.27 mW, (c) 0.30 mW, and (d) 0.33 mW

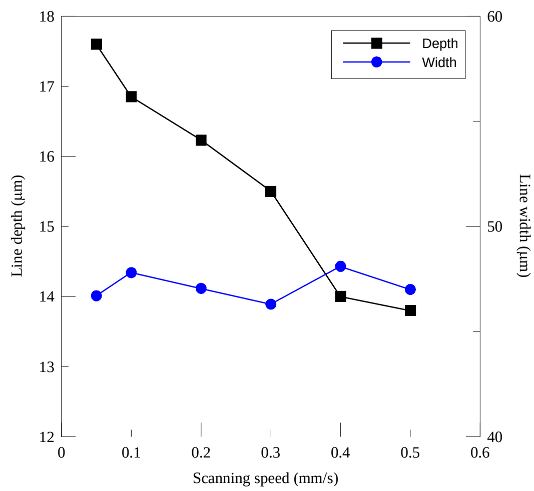


Fig. 5 Variation of line depth and width as a function of the scanning speed at a laser power of 0.122 mW.

Figure 6 is a plot of the variation of line depth and width as a function of the laser power at a scanning speed of 0.05 mm/s. It was observed that line width was constant at about 52 μm, whereas the line depth increased with increasing laser power.

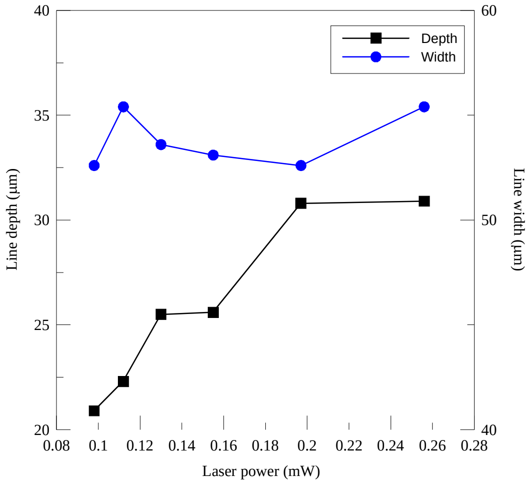
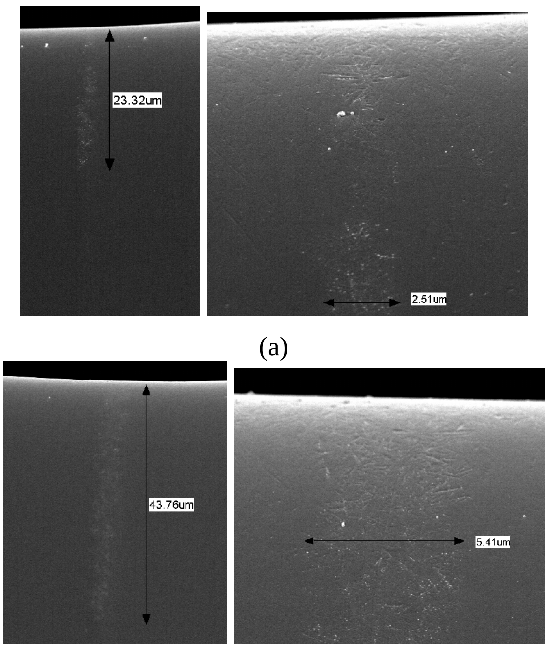


Fig. 6 Variation of line depth and width as a function of the laser power at a scanning speed of 0.05 mm/s.

It is necessary to gain an understanding of how crystallization is related to etching depth and width at different laser powers. Figure 7(a) and (b) presents cross-section SEM images of the line patterns fabricated at a scanning speed of 0.05 mm/s at laser powers of 0.18 mW and 0.23 mW, respectively. Note that the specimen is in a heated and unetched condition. As laser power increased, crystallization depth and width also increased. The crystallization width in Fig. 7(b) is about 5.41μm, which is much smaller than line width of 45μm obtained after etching, as shown in Fig. 4(a).



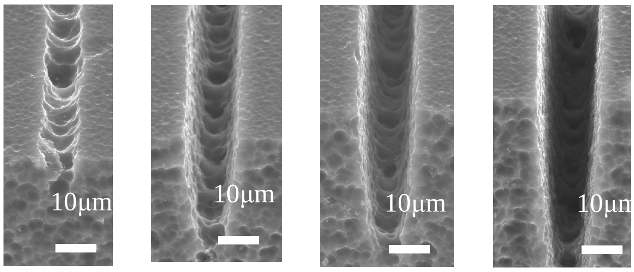
(a)

(b)

Fig. 7 Cross-section SEM images of irradiated Foturan glass after heat treatment and before etching: (a) 0.18 mW and (b) 0.23 mW.

Figure 8 shows the resulting line patterns after etching for reduced a reduced duration of 3 min. The SEM images presented show the line patterns fabricated under a scanning speed of 0.25 mm/s at four different laser powers, i.e., 0.3, 0.45, 0.6, and 0.75 mW, from which it can be seen that the line width was reduced to about 10 μm.

A schematic illustration of the 3D microchannel structure and the fabrication results is shown in Figs 9 and 10, respectively. The 3D microchannel structure shown was written at a laser power of 0.3 mW and a scanning speed of 0.05-mm/s. The sample was then etched for 3 hr. Figure 10(b) presents microscope images showing that cells had passed through the microchannel. These structures could be used in biomedical devices for purposes such as cell detection in the future.



(a) (b) (c) (d)

Fig. 8 SEM images of irradiated Foturan glass after heat treatment and etching at four laser powers: (a) 0.3 mW, (b) 0.45 mW, (c) 0.6 mW, and (d) 0.75 mW

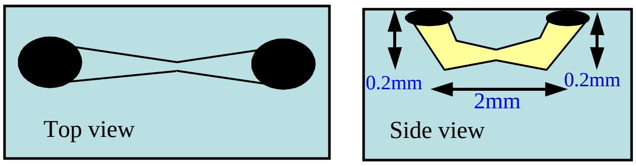
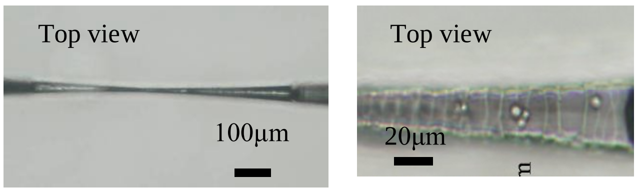


Fig. 9 Schematic illustration of a 3D microchannel structure.



(a) (b)

Fig. 10 Microscope images of the fabricated channel structure (a) before and (b) after cells had passed through the microchannel.

4. Conclusions

It was demonstrated in this study that etching time is an important parameter in the fabrication of microstructures on photosensitive glass by a femtosecond laser-induced crystallization process. The experimental results showed that microstructures can be fabricated on the surface of and inside the glass by careful control of the laser irradiation parameters and etching time. Overall, the proposed technique enables the fabrication of microstructures on photosensitive glass without etching of the surrounding area. A 3D microchannel structure was fabricated inside the glass, which could be applied in the fields of biomedicine, micro-fluidics, and cell detection in the future

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